PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Bitumen Composition of improved Temperature Susceptibility

We, ESSO RESEARCH AND ENGINEERING COMPANY, a Corporation duly organised and existing under the laws of the State of Delaware, United States of America, of Elizabeth, New Jersey, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to bitumen materials. In general, it relates to compositions which comprise a combination of an oxidized solvent extract and a vacuum reduced residuum, which compositions possess unusual characteristics making the composition eminently suitable, for example, as asphalt shingle sealants. Further, this invention relates to a process for preparing these compositions. In particular, the present invention concerns a composition suitable as a sealant for asphalt shingles, which composition comprises a combination of an air-oxidized phenol extract and a vacuum reduced aromatic residuum.

Bitumen, i.e., asphalt or pitch, compositions of high temperature susceptibility are useful in many applications. One such application is as a sealant for asphalt shingles. In such an application, the composition must meet rigid specifications; the most important of these specifications are set forth in Table I.

TABLE I

	Desired Specifications		
	Minimum	Typical	Maximum
Flash Point, °F. (COC*)	500	—	—
Softening Point °F.	165	—	187
Penetration at 77° F.	—	2.5	—
Penetration at 115° F.	12	—	17
Soluble in CCl_4 (wt. %)	99.5	—	—
Specific gravity	1.04	—	—
Viscosity, Saybolt Furol @ 400° F.	—	—	80

*COC = Cleveland Open Cup (A.S.T.M.) Method.

Attempts in the prior art to produce a bitumen composition which meets all of the above specifications have resulted in failure in most instances; and where successful initially have not been successful in consistently producing such compositions on a commercial scale. For example, attempts to produce a composition of the above-described desired specifications

from petroleum residua, e.g., a reduced crude, an aromatic tar from steam cracking, and catalytic fractionator bottoms after oxidation or straight reduction have proved fruitless since the compositions do not possess the required high temperature susceptibility. All such compositions are too brittle at 115°F. A composition which does meet the above specification has in the prior art been produced by air-blowing a lubricating oil extract (i.e., the solvent extract of a lube distillate) in order to obtain a composition which meets the softening point and penetration requirements; however, the composition produced by this latter process has not proved to be an acceptable process on a commercial basis. For instance, refinery data have shown that only rarely can a straight lube extract be air-blown to sufficient hardness at 500°F., the normal oxidation temperature. When the product can be made, the time required for air-blowing is uneconomically long; such as above 10 hrs.

It has now been found and forms the basis of this invention that a composition meeting the specifications set forth in Table I can be prepared by combining an oxidized solvent extract from a lubricating oil fraction of a naphthenic base oil (hereinafter called an oxidized solvent extract) and a vacuum reduced aromatic hydrocarbon distillation residuum (hereinafter called a vacuum reduced aromatic residuum) in certain proportions.

The oxidized solvent extract suitable for use in this invention is derived from a lube oil distillate. The lube oil distillate must be from a naphthenic base and will in general have a viscosity in the range of 75 to 150 SSU at 210°F. Paraffinic extracts are not compatible with aromatic residua since they precipitate asphaltenes from the latter. The solvent extract from which the oxidized solvent extracts of the present invention are produced are obtained from the solvent extraction of a lubricating oil distillate with a suitable solvent such as phenol. The solvent preferentially dissolves aromatic type components. The solvent extract is subsequently oxidized, for example by air-blowing, in order to produce the oxidized solvent extract useful in the present invention. The preparation of oxidized solvent extracts is well known to those skilled in the art. The oxidized solvent extract suitable for use in this invention will have the following specifications:

TABLE II

Inspections of Oxidized Solvent Extract

	Broad Range		
		Preferred Range	
Gravity °API	10	6 — 0.7	—3
Sp. Gr. 60/60°F.	1.00	1.03—1.07	1.10
Flash, °F. (COC)		500+	
C.C.R. %*	5	10—20	30
Sulphur, wt. %	1.5	2—4	4.5
Softening Point °F.	50	90—130	150

* Conradson Carbon Residue.

A preferred oxidized solvent extract suitable for use in this invention is one which is prepared by air oxidizing a phenol extract of a low cold temperature distillate having a viscosity in the range of 102 to 107 at 100 °F. and has the following typical inspections:

TABLE III

Inspections of 120° F. Softening Point Oxidized Phenol Extract*

Specific Gravity	1.05
Flash COC° F.	500+
Sulphur, wt. %	3.5
C.C.R %	16

* Prepared by air-blowing for 4.5 to 5.5 hrs. at 500° F. at 0.08 cu. ft./hr. This laboratory procedure correlates well with commercial practice.

The above described 120°F. Softening Point material is preferred because of its compatibility with heavy aromatics and also because of its commercial availability.

The vacuum reduced aromatic residua useful in the present invention may comprise the bottoms from a catalytic cracker or aromatic tar from steam cracking. In general, the vacuum reduced aromatic residua will have the following typical inspections:

TABLE IV

Inspections of Vacuum Reduced Aromatic Residua

	Aromatic Tar from Steam Cracking (Vacuum reduced)	Bottoms from Catalytic Cracker (Vacuum reduced)
Softening Point, °F.	300	250
Specific gravity 60/60° F.	1.18	1.25
Penetration at 77° F.	0.5	0.5
Penetration at 115° F.	1.0	1.0
Benzene Insol %	4	3
Asphaltenes, %	62	45
Aromatics and Polar Compounds %	38	55
Saturates %	Trace	Trace
Coking Value* %	55	50
Boiling Range, °F.	900+	975+

* Related to C.C.R. — usually 5 to 10 points lower than C.C.R.; it is a measure of the ability of the stock to form a coke/binder residue.

A preferred vacuum reduced aromatic residuum is one which is prepared by distillation of aromatic tar (from steam cracking) at reduced pressure, 30—100 mm Hg, to give overhead distillate, a sidestream and a bottoms fraction. This is essentially a flashing operation or perhaps 1 theoretical plate efficiency. The bottoms portion consists of 30—45 wt. % of the feed tar and is a hard resinous pitch of 250—350°F. softening point. It has the following typical inspections:

Softening Point, °F.	300
Specific gravity 60/60° F.	1.18
Penetration at 77° F.	0.5
Penetration at 115° F.	1.0
Benzene insoluble %	4
Asphaltenes, %	62
Aromatics and Polar Compounds %	38
Saturates %	trace
Coking Value* %	55
Boiling Range, °F.	900+

* Related to C.C.R. — usually 5 to 10 points lower than C.C.R.

The alternative to aromatic tar as feed to the vacuum still is catalytic fractionator bottoms. Under the same conditions as above, 20—30% bottoms is produced, of 225—300°F. softening point and is a pitch very similar to that from tar. The two pitches are interchangeable in the present invention. The oxidized solvent extract is a "soft pitch" and the vacuum reduced aromatic residuum is a "hard pitch." The softening points of both can be varied within a range of about 50—150°F. and 225—350°F. respectively without seriously affecting the invention, but adjustments in the percentages of each component will be required to compensate.

In general, the composition of the present invention comprises a combination of 60—75, preferably 64—69 wt. % oxidized solvent extract and 25—40, preferably 31—36 wt. % vacuum reduced aromatic residua. A preferred composition is a combination comprising 31—36 wt. % of a vacuum reduced aromatic residuum having about 300°F. softening point (Ring and Ball method) and 64—69 wt. % of an air-blown phenol extract having about 120°F. softening point.

EXAMPLE 1

1200 grams of an oxidized solvent extract was prepared by air-blowing 1300 grams of a phenol extract with an air flow rate of 0.08 cubic feet per hour for a period of 4½ to 5½ hours and at a temperature of 500°F. The phenol extract used was prepared by phenol treating a low cold test distillate having a viscosity in the range of 102—107 SUS at 210°F. and had the following typical inspections.

Aniline Point, °F.	89
C.C.R., %	3.2
Flash COC° F.	500+
Gravity °API	7.0
Pour Point, °F.	65
Sulphur, wt. %	3.3
Viscosity SUS @ 210° F.	270

The air-blown phenol extract had a softening point of 120°F. This product is Oxidized Solvent Extract A.

EXAMPLE 2

Oxidized Solvent Extract B was prepared in a manner identical to that used in Example 1 with the exception that the air-blowing lasted for a period of 3½ to 4½ hours yielding a product which had a softening point of about 99°F.

EXAMPLE 3

A vacuum reduced aromatic residuum was prepared by distillation of aromatic tar, from steam cracking, at reduced pressure of 30—100 mm Hg, to give an overhead distillate, a sidestream and a bottoms fraction. The bottoms fraction consisted of 30—45 wt. % of the feed and was a hard resinous pitch of 250—350°F. Softening Point. This product was Vacuum Reduced Aromatic Residuum Z.

In order to demonstrate the efficacy and the utility of the compositions of the present invention, several bitumen compositions were prepared including compositions utilizing the products of Examples 1—3 above. The properties of these compositions were determined and compared to the desired properties as defined by the specifications set forth in Table I. These compositions and their properties are set forth in Table V.

The above data show that only with the composition of the present invention comprising a combination of certain proportions of an oxidized solvent extract and a vacuum reduced aromatic residua can all the specification requirements be fulfilled. All compositions prepared from other components failed, in one or more respects, to meet the required specifications.

TABLE V
Bitumen Compositions

Formulation	Composition, wt. %				Properties		
	Solvent Extract	Oxidized Solvent Extract	Residua	Vacuum Reduced Aromatic Residua	Softening Point, °F.	Penetration, 77° F.	Penetration, 115° F.
D	—	—	100 (1)	—	172	1.0	2.0
E	—	—	55 (2)	45 (3)	172.5	1.0	10.0
F	50 (4)	—	—	50 (3)	—	—	—
G	59 (5)	—	—	41 (3)	170	9.0	22.0
H	55 (5)	—	—	45 (3)	197	2.0	8.0
J	57 (5)	—	—	43 (3)	163	12.5	35.0
K	—	70 (6)	—	30 (3)	174	7.0	20.0
L	—	64 (6)	—	36 (3)	186	2.5	10.0
M	—	66 (6)	—	34 (3)	184	4.0	11.0
N	—	67.5	—	32.5 (3)	178.5	3.5	14.0
O	—	60 (7)	—	40 (3)	196	2.0	6
P	—	65 (7)	—	35 (3)	179	5.0	17
Q	—	63 (7)	—	37 (3)	190	3.0	11

(1) Partially reduced Catalytic Fractionator Bottoms, 950° F. + Bottoms.
(2) Partially reduced Catalytic Fractionator Bottoms, 850° F. + Bottoms, softening point below room temperature.
(3) From Example 3.
(4) Phenol extract from treating Paraffin Distillate 180/190 and having a viscosity of 1000 SUS @ 210, Pour Point of 105° F., Gravity of 3.5° API, Flash of 530+°F., C.C.R. of 10.8% and an Aniline Point of 140.
(5) Phenol extract of Examples 1 and 2.
(6) From Example 1.
(7) From Example 2.

WHAT WE CLAIM IS:—

1. A bitumen composition comprising in combination 25 to 40 wt. % of a vacuum reduced aromatic hydrocarbon distillation residuum and 60 to 75 wt. % of an oxidized solvent extract of a naphthenic base lubricating oil fraction.

2. A composition as claimed in claim 1 wherein said oxidized solvent extract is an air-oxidized phenol extract having a softening point in the range 50 to 150°F.

3. A composition as claimed in claim 1 or claim 2 wherein said vacuum reduced aromatic residuum has a softening point in the range of 225 to 350°F.

4. A bitumen composition as claimed in any of the preceding claims comprising in combination 64 to 69 wt. % of an oxidized solvent extract and 31 to 36 wt. % vacuum reduced aromatic residuum.

5. A composition as claimed in claim 4 wherein said oxidized solvent extract has a softening point of 90 to 130°F. and wherein said vacuum reduced aromatic residuum has a softening point of about 300°F.

6. A composition as claimed in claim 4 or claim 5 wherein said oxidized solvent extract is an air oxidized phenol extract having a softening point of about 120°F.

7. A composition as claimed in claim 1 and substantially as hereinbefore described.

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